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Indian Standard

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फॉर्मिक एसिड की विशिष्टि

(पहला पुनरीक्षण)

Specification for Formic Acid

(First Revision)

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Organic Chemicals, Alcohol and Allied Products Sectional Committee, PCD 09

FOREWORD

This Indian Standard (First revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Organic Chemicals, Alcohol and Allied Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was originally published in 1981 by taking assistance from BS 4341 : 1968 'Specification for formic acid', ANALAR standards for laboratory chemicals and ISO/R 731-1968 'Formic acid for industrial use — Methods of analysis'.

The Committee, responsible for revision of the standard, has observed that, now a days more sophisticated instruments having improved minimum detection limits are available for testing for iron. Considering this, the committee decided to revise the standard. In this revision alternate test methods for iron has been added and Amendment no. 1 and 2 have also been incorporated along with some other minor modifications.

Formic acid is used in the manufacture of nickel format, fumigants, insecticides, refrigerants and various other chemicals. It is also used as solvent for perfumes, lacquers, in tanning, and wine manufacture to aid fermentation, medicines, brewing, food preservative, silvering glass, leather, rubber coagulant, electroplating, ore floatation and dyeing and finishing of textiles and paper and for analytical purposes.

The composition of the Committee, responsible for the formulation of this standard is given at Annex C.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

SPECIFICATION FOR FORMIC ACID

(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for formic acid.

2 REFERENCES

The following standards contain provisions which through reference in this text, constitute provisions of the standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on these standards are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
265 : 1993	Hydrochloric acid — Specification (*fourth revision*)
915 : 2012	Laboratory glassware — One-mark volumetric flasks (*third revision*)
1070 : 1992	Reagent grade water — Specification (*third revision*)
4161 : 1967	Specification for Nessler cylinders
4905 : 2015	Random sampling and randomization procedures (*first revision*)
6312 : 1994	Polyethylene containers for the transport of materials — Specification (*second revision*)

3 GRADES

There shall be two grades of the material, namely:

a) *Grade 1* — Generally used for the manufacture of camphor, and

b) *Grade 2* — For industrial purpose.

4 REQUIREMENTS

4.1 The material shall be clear, colourless, volatile and pungent smelling liquid, miscible in water and alcohol in all proportions.

4.2 The material when tested according to the methods given in Annex A shall also comply with the requirements given in Table 1. Reference to the relevant clauses of Annex A is given in Table 1.

5. PACKING AND MARKING:

5.1 Packing

Unless otherwise agreed to between the purchaser and the supplier, the material shall be packed in narrow neck flasks with ground glass stopper or suitable plastic lid with threads. Large quantities are supplied in plastic or glass or high density polyethylene carboys (*see* IS 6312). The containers shall be kept in a suitable place at ambient condition under shed.

Table 1 Requirements for Formic Acid

(*Clause* 4.2)

Sl No.	Characteristic	Requirement		Method of Test (Ref to Cl No. in Annex A)
		Grade1	Grade2	
(1)	(2)	(3)	(4)	(5)
i)	Total acidity (as HCOOH), percent by mass, *Min*	90	85	A-3
ii)	Water insoluble matter	To pass test	To pass test	A-4
iii)	Non-volatile matter, percent by mass, *Max*	0.06	0.08	A-5
iv)	Acids other than formic, percent by mass, *Max*	1.0	1.0	A-6
v)	Chlorides (as Cl), percent by mass, *Max*	0.006	0.005	A-7
vi)	Nitrogen compounds (as N), percent by mass, *Max*	-	0.002	A-8
vii)	Sulphates (as SO4), percent by mass, *Max*	0.03	0.03	A-9
viii)	Iron (as Fe), percent by mass, *Max*	0.005	0.005	A-10
ix)	Heavy metals (as Pb), percent by mass, *Max*	0.002	0.002	A-11

5.2 Marking

5.2.1 Each container shall be securely closed and shall bear legibly and indelibly the following information:

a) Name and grade of the material;
b) Name of the manufacturer and his recognized trade-mark, if any
c) Net mass of the material;
d) Lot or batch number in code or otherwise; and
e) The year of manufacture.

5.2.2 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

6 SAMPLING AND CRITXERIA FOR CONFORMITY

Representative samples of the material shall be drawn and their conformity to the standard determined as prescribed in Annex B.

ANNEX A

(*Clause* 4.2, and *Table* 1)

METHODS OF TEST FOR FORMIC ACID

A-1 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* **IS 1070)** shall be employed in tests.

NOTE —Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 SAMPLE FOR TEST

The laboratory sample should be of sufficient size for carrying out all the analysis. It should be preserved in an airtight glass-stoppered bottle of such a size that it is nearly filled by the sample. If it is considered necessary to seal the container, care should be taken to avoid the risk of contaminating the contents in any way.

A-3 ASSAY

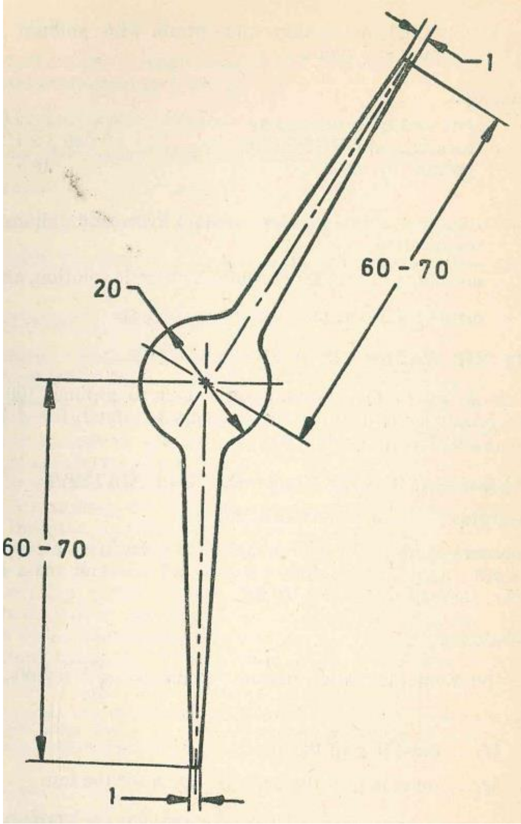
A-3.0 Principle

Titration of acidity using a standard volumetric solution of sodium hydroxide using phenolphthalein as indicator.

A-3.1 Apparatus

A-3.1.1 Flat-bottom weighing bottle with lid, diameter approximately 20 mm or equivalent any other suitable device. This is only required for samples believed to contain less than 90 percent (*m/m*) of total acidity that is, for Grade 2.

A-3.1.2 Glass ampoule (*see* Fig. 1).



All dimensions millimeters

FIG. 1 GLASS AMPOULES

A-3.2 Reagents

A-3.2.1 Standard Sodium Hydroxide Solution — *1 N or different normality with appropriate sample weight.*

A-3.2.2 *Phenolphthalein Indicator* — Dissolve 0.5 g of phenolphthalein in 100 ml of 80 percent (v/v) rectified spirit and make faintly pink by the addition of dilute sodium hydroxide solution.

A-3.3 Procedure

A-3.3.1 *For Acidity More than 90 Percent (as Formic Acid)*

Weigh the glass ampoule (*see* Fig. 1) to the nearest 0.5 mg and introduce a sample about 1.5 g by partially immersing the ampoule in the bottle containing the material. Seal the capillary without loss of glass, using the oxidizing flame of the burner, withdraw the ampoule and invert it. Wipe the end that was immersed in the laboratory sample with a filter paper and seal rapidly. Weigh the ampoule again to the nearest 0.5 mg and determine to the nearest milligram by difference between the two weighing, the mass of sample taken.

Place the sealed ampoule in a 250 ml conical flask containing about 50 ml of water. Break the ampoule with a glass rod, shake, add 0.5 ml of phenolphthalein solution and titrate with sodium hydroxide solution to a faint pink end point.

A-3.3.2 *For Acids Containing Less than 90 Percent (m/m) of Total Acidity, Expressed as Formic Acid*

Take 1.5 to 2.0 g of the test portion into the flat-bottom weighing bottle and weigh to the nearest milligram. Transfer into a 250 ml conical flask containing about 50 ml of water. Add 0.5 ml of phenolphthalein solution and titrate with sodium hydroxide solution to a faint pink end point.

A-3.4 Calculation

$$\text{Total acidity, expressed as formic acid percent by mass} = \frac{0.0463 \times V \times N}{M}$$

Where,

V = volume in ml, of standard sodium hydroxide solution used for the titration;

N = normality of standard sodium hydroxide solution; and

M = mass in g, of the material taken for test.

A-4 TEST FOR WATER INSOLUBLE MATTER

Dilute 25 ml of the material with water to produce 100 ml in glass beaker or conical jar and allow the contents to stand for 1 h. The solution shall be clear and colourless.

A-5. DETERMINATION OF NON-VOLATILE MATTER

A-5.1 Apparatus

Basin, porcelain or silica or borosilicate glass.

A-5.2 Procedure

Take 10 g of material in a basin and evaporate it on a water bath. Dry the residue at 105°C till constant mass is obtained. Preserve the residue for test in **A-10.3**.

A-5.3 Calculation

$$\text{Non-volatile matter, percent by mass} = \frac{M_1}{M_2} \times 100$$

Where,

M_1 = mass in g, of the residue; and

M_2 = mass in g, of the material taken for the test.

A-6 DETERMINATION OF ACIDS OTHER THAN FORMIC ACID

A-6.0 Field of Application

This method is applicable to formic acid, containing between 0.5 and 6.0 percent (*m/m*) of other acids calculated as acetic acid.

A-6.1 Principle

Titration with sodium hydroxide solution, using a *pH* meter, of the acids remaining after quantitative oxidation of formic acid with excess mercuric oxide.

A-6.2 Reagents

A-6.2.1 *Acetic Acid,* approximately 0.5 percent (v/v) solution. Dilute 5 ml of glacial acetic acid to 1 000 ml.

A-6.2.2 *Mercuric Oxide (Red)*

A-6.2.3 *Standard Sodium Hydroxide Solution,* 0.1 N.

A-6.3 Apparatus

A-6.3.1 *Conical Flasks*, 250 ml capacity.

A-6.3.2 *Reflux Condenser*, water cooled, with ground glass joints to fit the flasks.

A-6.3.3 *pH Meter*, with a glass electrode.

A-6.4 Procedure

Place 5 g of the material for acids less than 2 percent and 2 g of the material for acids more than 2 percent in one of the conical flasks. Add exactly 5.0 ml of acetic acid solution and an amount of mercuric oxide. Calculate on the basis of 5.5 g of mercuric oxide per gram of formic acid present in the test portion. Then add sufficient water to bring the total volume to about 30 ml. Fit the flask with a condenser and heat gently for 10 min. Fairly strong evolution of carbon dioxide from the solution containing the test portion occurs. Reflux gently for 30 min and rinse the condenser with 20 to 25 ml of water. Cool the flask to room temperature and pour the contents without filtering into a 250 ml beaker. Rinse the flask into the beaker with 20 to 25 ml of water, and transfer the washings to the beaker. Stir the solution vigorously, preferably using a magnetic stirrer, and keep stirring during the titration. Titrate the residual acids with sodium hydroxide solution using the *pH* meter. Similarly carry out a blank titration. The end point of the titration is at *pH* 8.6 for the test and at *pH* 8.3 for the blank.

NOTE — Samples, requiring more than 10 to 15 ml of sodium hydroxide solution do not give a very sharp end point, will need vigorous stirring for longer period in order to obtain a stable final *p*H.

A-6.5 Calculation

Acids other than formic acid,

$$\text{percent by mass} = \frac{(V_1 - V_2) \times 0.0060 \times N}{M}$$

Where,

V_1 = volume in ml, of standard sodium hydroxide solution used for the titration of the material;

V_2 = volume in ml, of standard sodium hydroxide solution used for the titration of the blank;

N = normality of standard sodium hydroxide solution; and

M = mass in g, of the material taken for the test.

A-7 TEST FOR CHLORIDE

A-7.0 Outline of the Method

The material is dissolved in water and acidified with nitric acid. Silver nitrate is then added and turbidity compared with that produced by a known quantity of chloride.

A-7.1 Apparatus

A-7.1.1 *One-Mark Volumetric Flasks* — 1 000 ml (*see* IS 915).

A-7.1.2 *Nessler Cylinders* — 50 ml (*see* IS 4161).

A-7.2 Reagents

A-7.2.1 *Dilute Nitric Acid* — approximately 5 N.

A-7.2.2 *Silver Nitrate Solution* — 10 percent (*m/v).*

A-7.2.3 *Standard Sodium Chloride Solution* — Dissolve 1.65 g of sodium chloride in water and transfer quantitatively to one of the 1 000 ml one mark volumetric flasks. Make up the volume with water to the mark. Pipette out 10 ml of this solution into the second one-mark volumetric flask and again dilute with water to 1 000 ml mark. One milliliter of this solution contains 0.01 mg of chloride.

A-7.3 Procedure

A-7.3.1 *For Grade 2* — Dissolve 2-0 g of the material in 50 ml of water and add 5 ml of dilute nitric acid and 1 ml of silver nitrate solution.

A-7.3.1.1 The material shall be taken to have not exceeded the limit prescribed if no opalescence is produced.

A-7.3.2 *For Grade* 1 — Dissolve 1.00 g of the material in 30 ml of water and transfer this solution quantitatively to one of the Nessler cylinders. Add 5 ml of dilute nitric acid and 1 ml of silver nitrate solution. Dilute with water to 50 ml mark and mix well. In the second Nessler cylinder carry out a control test under the same conditions using 5 ml of standard chloride solution in place of the material. Dilute the contents with water to 50 ml mark and mix well.

A-7.3.2.1 The material shall be taken to have not exceeded the limit prescribed if the opalescence or turbidity produced with the material is not greater than that obtained in the control test.

A-8 TEST FOR NITROGEN COMPOUNDS

A-8.1 Apparatus

A-8.1.1 *Nessler Cylinders*, 50 ml capacity.

A-8.1.2 Hessian Crucible or any other Crucible, suitable for the purpose.

A-8.1.3 Micro-Kjeldahl Distillation Apparatus.

A-8.2 Reagents

A-8.2.1 *Devarda's Alloy (Containing 45 Parts Aluminium, 50 Parts Copper and 5 Parts Zinc*)

Heat the aluminium in a Hessian crucible in a furnace until the aluminium begins to melt. Add copper in small portions and heat until liquefied. Now plunge zinc into the molten mass. Cover the crucible and heat the mixture for a few minutes and stir with a clean iron rod. Allow it to cool slowly with the cover on and pulverize the crystallized mass. Alternatively, readily available Devarda's alloy AR grade may be used.

A-8.2.3 *Nessler Solution*

Dissolve 10 g of potassium iodide in 10 ml of ammonia-free water, and add to it slowly, while stirring, saturated mercuric chloride solution until a slight permanent precipitate is formed. Add 30 g of potassium hydroxide and, when it has dissolved, add 1 ml more of saturated mercuric chloride solution and dilute to 200 ml with ammonia-free water. Allow to settle overnight, decant the clean solution and keep it in a bottle with a well-fitting rubber stopper.

A-8.2.4 *Standard Nitrogen Solution*

Dissolve 0.381 9 g of ammonium chloride in enough water to make up the volume to 1 000 ml. Pipette out 10 ml from this solution and dilute with water to 100 ml. One milliliter of this solution is equivalent to 0.01 mg of nitrogen.

A-8.3 Procedure

A-8.3.1 *For Grade* 2

Dilute 1.000 g of the material in 10 ml water. Transfer 5 ml of this solution to a micro-Kjeldahl distillation apparatus and add 25 ml of sodium hydroxide solution, nitrogen-free, and 0.5 g Devarda's alloy. Heat or pass steam into the distillation flask and collect any distillate in 5 ml of water containing 1 ml of dilute hydrochloric acid. Continue to pass the steam until the liquid boils; disconnect the steam, allow the reaction to proceed for one hour and then steam-distil until a total volume of 50 ml is obtained during about 6 min. Prepare a standard in the same manner using 2 ml of standard nitrogen solution in place of the 5ml aliquot of sample solution. To each distillate add 2 ml of sodium hydroxide solution, ammonia-free, and 2 ml of Nessler's solution.

A-8.3.1.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced in the test with the material is not greater than that produced in the control test.

A-9 TEST FOR SULPHATE

A-9.0 Outline of the Method

The material is dissolved in water, denatured spirit and dilute hydrochloric acid added and mixed. Barium chloride solution is then added and turbidity compared with that produced by a known quantity of sulphate.

A-9.1 Apparatus

A-9.1.1 *One-Mark Volumetric Flasks* — 100 ml and 1 000 ml (*see* IS 915).

A-9.1.2 *Nessler Cylinders* — 50 ml (*see* IS 4161).

A-9.2 Reagents

A-9.2.1 *Denatured Spirit*

A-9.2.2 *Dilute Hydrochloric Acid* — approximately 5 N.

A-9.2.3 *Barium Chloride* ($BaCl_2$ $2H_2O$) *Solution* — 10 percent (m/v).

A-9.2.4 *Standard Sulphate Solution* — Dissolve 1.48 g of ignited sodium sulphate (Na_2S0_4) in water. Transfer quantitatively to the 1 000 ml one mark volumetric flask and make up the volume with water to the mark. Pipette 10 ml of this solution into the 100 ml one-mark volumetric flask and make up the volume with water to the mark. One milliliter of this solution contains 0.1 mg of sulphate (as SO_4).

A-9.3 Procedure

Weigh 1.000 g of the material for grade 1 and 10.000 g for grade 2 and to this add 0.2 ml of sodium carbonate, Evaporate the contents to dryness on a steam bath. Dissolve the residue in 40 ml of water, add 10 ml of denatured spirit and 1 ml of dilute hydrochloric acid; mix and add 1 ml of barium chloride solution. Mix immediately and allow to stand for 1 h. Similarly carry out a control test under same conditions and using 1 ml of standard sulphate solution in place of the material.

A-9.3.1 The material shall be taken to have not exceeded the limit prescribed for grade 1 and grade 2 if the turbidity produced with the material is not greater than that obtained in the control test.

A-10 DETERMINATION OF IRON

A 10.1 – Method A

A-10.1.0 *Outline of the Method*

In an alkaline medium, thioglycolic acid gives a reddish violet colouration with ferrous and ferric iron. The residue for the ash determination is dissolved in hydrochloric acid, thioglycolic acid added and then made alkaline by the addition of ammonia. The colour developed is then compared with that obtained with standard iron solution similarly treated. The iron content is then determined from the amount of standard iron solution required for matching the colour.

A-10.1.1 *Apparatus*

A-10.1.1.1 *One-mark volumetric flasks* — 1 000 ml capacity (*see* IS 915) .

A-10.1.1.2 *One-mark volumetric flasks* — 25 ml capacity (*see* IS 915).

A-10.1.1.3 *Nessler cylinders* — 100 ml capacity (*see* IS 4161).

A-10.1.2 *Reagents*

A-10.1.2.1 *Hydrochloric acid* — Relative density 1.16 (*see* IS 265)

A-10.1.2.2 *Ammonium hydroxide* — Relative density 0·88.

A-10.1.2.3 *Thioglycolic acid solution* — 10 percent (v/v).

A-10.1.2.4 *Citric acid solution* — 30 percent (*m/v*).

A-10.1.2.5 *Standard iron solution* — Dissolve 0.702 2 g of ammonium ferrous sulphate [(NH4) $_2$S0$_4$ FeS0$_4$6H$_a$O)] in 50 ml of dilute sulphuric acid and then transfer to one of the 1 000 ml one-mark graduated flasks. Dilute with water to 1 000 ml mark. Pipette out 100 ml of this solution into the second 1 000 ml one-mark graduated flask. Dilute 100 ml of this solution again to 1 000 ml. One milliliter of this solution contains 10 mg of iron.

A-10.1.3 *Procedure*

To the basin containing the residue from the determination of non-volatile matter (*see* **A-5.1**), add 5 ml of concentrated hydrochloric acid. Heat the basin on a boiling water-bath, agitating with a stirrer or platinum wire until all the residue has dissolved. Allow to cool, transfer to the 25 ml volumetric flask, dilute to the mark with water and mix thoroughly. For each determination, transfer 10 ml of this solution to a 100 ml Nessler cylinder, dilute to about 30 ml and add 0.5 ml of citric acid solution followed by 1 ml of thioglycolic acid solution.

Add ammonium hydroxide carefully until a reddish purple colour just appears and then add 0.5 ml in excess. Dilute to 100 ml and mix thoroughly. To about 90 ml of water in the second 100 ml Nessler cylinder add 2 ml of the concentrated hydrochloric acid, then 0.5 ml of the citric acid solution followed by 1 ml of the thioglycolic acid solution and 3 ml of the ammonium hydroxide. Add the standard iron solution slowly from a burette shaking with each addition, until the depth of colour in the two cylinders is identical when they are viewed along their axes. Record the volume of standard iron solution added.

A-10.1.4 *Calculation*

Iron content (as Fe), parts per million, by mass = $\frac{25V}{M}$

Where,

V = volume in ml, of standard iron solution used; and

M = mass in g, of sample taken for the determination of non-volatile matter (**A-5.1**).

A 10.2 Method B

A-10.2.0 *Outline of the Method*

Iron (Fe^{+2}) reacts with 1,l0-phenanthroline to form an orange-red complex $[(C_{12},H_8N_2)_3Fe]^{2+}$.The colour intensity is independent of the acidity in the pH range 2-9, and is stable for long periods. The complex which follows Beers's law closely and its absorbance is very stable for long period.

Ferric iron reduced with hydroquinone and pH is adjusted with an acetate buffer. The solution, slightly acid with hydrochloric acid, is treated with1, l0 phenanthroline, and buffered with sodium citrate at a pH of 4.The iron - phenanthroline complex is measured at 510 nm against reagent blank.

A-10.2.1 *Apparatus*

A-10.2.1.1 *One-mark volumetric flasks* — 1 000 ml capacity (*see* IS 915).

A-10.2.1.1 *One-mark volumetric flasks* — 500 ml capacity (*see* IS 915).

A-10.2.1.2 *One-mark volumetric flasks* — 50 ml capacity (*see* IS 915).

A – 10.2.1.3 *UV-Vis spectrophotometer*

A-10.2.1.4 *Glass cell of 50 mm path length* — 2 Nos.

A-10.2.1.5 *Standard micro burette 10 ml capacity.*

A-10.2.2 *Reagents*

A-10.2.2.1 *Hydrochloric acid* — relative density 1.16 (*see* IS 265)

1 : 1 HCl : Dilute hydrochloric acid with equal volume of DM water.

A-10.2.2.2 One percent hydroquinone solution in DM water. Kept in brown bottle and discard when turbid.

A-10.2.2.3 25 percent of sodium citrate solution in DM water

A-10.2.2.4 0.10 percent of 1, 10 phenanthrolinein DM water

A-10.2.2.5 *Standard iron solution* — Dissolve 3.511grm of ammonium ferrous sulphate ($FeSO_4$ $(NH_4)2SO_4$, $6H_2O$) in 500 ml DM water containing 10 ml con H_2SO_4. 1 ml = 1 000 micro gram iron -----> (A).

Take 2 ml of soln. (A) with micro burette in 1 000 ml volumetric flask. Make up with DM water. It will give 1 ml = 2 micro gram -------> (B)

A-10.2.3.1 *Procedure*

To the basin containing the residue from the determination of non-volatile matter (*see* **A-5.1**), add 5 ml of concentrated hydrochloric acid. Heat the basin on a boiling water-bath, agitating with a glass rod until all the residue has dissolved. Allow to cool, transfer to the 50 ml volumetric flask, (if high iron content is expected, then make up the volume to 50ml and take suitable aliquot into another 50 ml volumetric flask). Add 1 ml 1:1 HCl, add 1.0 ml of 1 percent hydroquinone solution, stir well and wait for 5 min. Add 5 ml 0.1 percent 1, 10 phenanthroline and 5 ml 25 percent sodium citrate solution. Stir well and make up the volume up to the mark with DM water. Measure the absorbance or direct concentration after 30 min using 50 mm cell at 510 nm wavelength.

Calculation:

$$\text{Iron as Fe, ppm} = \frac{\text{Micro gm of Fe} \times \text{dilution factor if applicable}}{\text{M} \times \text{aliquot of sample stock}}$$

Where,

M = mass in g, of sample taken for the determination of non-volatile matter (**A-5.1**).

A-10.2.3.2 *Calibration of spectrophotometer for iron:*

Calibration Range: 0 to 10 Micro gram

Procedure

Take 1,2,3,4, and 5 ml of std. (B) (*see* **10.2.2.5**) in 50 ml volumetric flask keeping sixth as blank. Add 1 ml 1:1 HCl, add 1.0 ml of 1 percent hydroquinone solution, stir well and wait for 5 min. Add 5 ml 0.1 percent 1, 10 phenanthroline and 5 ml 25 percent sodium citrate solution. Stir well and make up the volume up to the mark with DM water. Measure the absorbance or direct concentration after 30 min using 50 mm cell at 510 nm wavelength. Measured absorbance data and slope value are mentioned.

NOTE:

1. Other type of spectrophotometric method may be used, provided slope determination for the specified range is established and accuracy level as specified in this standard is achievable.

2. For analysis of iron instrumental method of analysis like AAS, ICP may be used, provided calibration of instrument is done in accordance with detection levels as necessary with requirements of this standard.

A-11 TEST FOR HEAVY METAL CONTENT

A-11.0 Outline of the Method

The colour produced by heavy metals with hydrogen sulphide is compared to that produced by standard lead solution under identical conditions.

A-11.1 Reagents

A-11.1.1 *Dilute Acetic Acid* — 1:1. Dilute acetic acid which complies with the following test:

Evaporate 20 ml of the acid in porcelain dish nearly to dryness on a steam-bath. Add to the residue 2 ml of the acetic acid and dilute with water to 25 ml. Then add 10 ml of a solution of hydrogen sulphide. Any dark colour produced shall not be darker than a control made with 0.04 mg of lead and 1 ml of the diluted acetic acid (2 parts per million).

A-11.1.2 *Hydrochloric Acid* — Hydrochloric acid which complied with the following tests:

Evaporate 17 ml of the acid in a beaker to dryness on a steam-bath. Dissolve the residue in 2 ml of dilute acetic acid, dilute to 40 ml with water and add 10 ml of solution of hydrogen sulphide; any darkening produced shall not be greater than in a blank test to which 0.02 mg of lead has been added (1 part per million).

A-11.1.3 *Hydrogen Sulphide Solution* — A freshly prepared saturated solution of hydrogen sulphide in water.

A-11.1.4 *Stock Lead Nitrate Solution* — Dissolve 159.8 mg of lead nitrate in 100 ml of water to which 1 ml of nitric acid has been added, then dilute to 1 000 ml with water.

NOTE — This solution shall be prepared and stored in glass containers free from soluble lead salts.

A-11.1.5 *Standard Lead Solution* — Dilute 10 ml of the stock solution of lead nitrate accurately to 100 ml with water. This solution shall be freshly prepared. Each milliliter of this standard lead solution contains the equivalent of 0.01 mg of lead.

A-11.1.6 *Test Solutions*

Solution A — Introduce into a 50 ml Nessler cylinder 2 ml of dilute acetic acid and 2 ml of standard lead solution for grade 1 of the material and 0.5 ml for grade 2 and make up to 25 ml with water.

Solution B — Dissolve 1 g of the material under test in 10 ml of water, add 2 ml of dilute hydrochloric acid and water to make 25 ml.

A-11.2 Procedure

Transfer solutions A and B to matching 50 ml Nessler cylinders, add 10 ml of solution of hydrogen sulphide to each cylinder, mix, allow to stand for 10 min, then view downward over a white surface, the colour of solution B shall not be darker than that of solution A.

ANNEX B

(*Clause* 6.1)

SAMPLING OF FORMIC ACID

B-1 GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall be taken at a place protected from damp air, dust and soot.

B-l.2 Sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected, for sampling shall be mixed as thoroughly as possible by suitable means. While drawing samples, the sampler shall avoid breathing vapours of the material.

B-1.5 The samples shall be placed in clean, dry and air-tight glass or other suitable containers on which the material has no action.

B-1.6 The sample containers shall be of such a size that they are almost but not completely filled by the sample.

B-1.7 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling, year of manufacture, and other important particulars of the consignment.

B-2 SCALE OF SAMPLING

B-2.1 Lot — All containers in a single consignment of the same grade of material and belonging to the same batch of manufacture shall be grouped together and each such group shall constitute a lot.

B-2.2 For ascertaining the conformity of lot to the requirements of this specification samples shall be tested of each separately. The number of containers to be sampled shall depend on the size of the lot and shall be in accordance to Table 2.

B-2.2.1 These containers shall be selected at random. In order to ensure the randomness of selection, procedures in IS 4905 may be followed.

B-3 TEST SAMPLES AND REFEREE SAMPLE

B-3.1 From each of the containers selected in **B-2.2**, draw with an appropriate sampling instrument small portions of the material from different parts of the container. The total quantity so drawn from each of the containers shall be approximately equal to thrice the quantity required for testing purposes.

Table 2 Scale of Sampling

Lot Size	No. of Containers To Be Selected
Up to 15	3
16 - 50	4
51 - 100	5
101 - 150	6
161 - 300	7
301 - 500	8
601 - 1000	9
1001 and above	10

B-3.2 Mix thoroughly all the portions of the material drawn from the same container to give a representative sample for the container.

B-3.3 From the samples **(B-3.2)** representing different containers selected in **B-2.2,** a small but equal quantity of material shall be taken and thoroughly mixed to form a composite sample. The composite sample so obtained shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

B-3.4 The remaining portion of the material in the samples **(B-3.2)** from different containers shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the containers selected shall be for the purchaser, another for the supplier and the third for the referee.

B-3.5 All the individual and composite samples shall be transferred to separate containers. These containers shall then be sealed air-tight with stopper and labelled with full identification particulars given in **B-1.7.**

B-3.6 The referee samples consisting of a composite sample and a set of individual samples shall bear the seals of both the purchaser and the supplier and shall be kept at a place agreed to between the two. These shall be used in case of any dispute between the two.

B-4 NUMBER OF TESTS

B-4.1 The total acidity shall be tested on each of the individual samples.

B-4.2 The remaining characteristics given in Table 1 shall be tested on the composite sample.

B-5 CRITERIA FOR CONFORMITY

B-5.1 The lot shall be deemed as conforming to the requirements of total acidity if all test results on each of the individual samples satisfy the corresponding requirements given in Table 1.

B-5.2 For determining the conformity of the lot with respect to other requirements test results on the composite sample shall satisfy the corresponding requirements given in Table 1.

B-5.3 For declaring the conformity of the lot to the requirements of this specification, **B-5.1** and **B-5.2** shall be satisfied.

ANNEX C

(*Foreword*)

COMMITTEE COMPOSITION

Organic, Chemicals Alcohols and Allied Products Sectional Committee, PCD 09

Organization(s)	*Representative(s)*
Chemical Engineering and Process Development Division, NCL PUNE	Dr C. V. Rode (***Chairman***)
All India Distilleries Association (AIDA), New Delhi	Shri V. N. Raina
BASF India Limited, Mumbai	Shri Kiran Bhat Shri Hemal Berawala (*Alternate*)
CDRI, Lucknow	Dr Sanjeev Kanojiya
Central Revenues Control Laboratory, Delhi	Dr T. A. Sreenivasa Rao
Chemical and Petrochemicals Manufacturers Association (CPMA), New Delhi	Shri Kamal Nanavaty
Deepak Fertilizer, Pune	Dr Satish Chand Saini Shri Suresh Amle (*Alternate*)
Deepak Phenolics Limited, Bharuch	Dr S. Samal Shri N. R. Sandipkumar Pancha (*Alternate*)
Dow-Corning India Limited, Mumbai	Shri Abraham Barretto Shri Ritesh Gulabani (*Alternate*)
Indian Chemical Council, Mumbai	Dr Mritunjay Chaubey Shri J. I. Sevak (*Alternate*)
Jubilant Life Sciences Ltd, Noida	Shri Hari Mohan Lohani
National Chemical Laboratory, Pune	Dr Udaya Kiran Marelli
UPL Limited, Mumbai	Mr M. D. Vachhani, Gm (Qa)
Alkyl Amines Chemicals Ltd, Mumbai	Shri S. V. Nikumbhe Shri Sameer Katdare (*Alternate*)
All India Alcohol-Based Industries Development Association (AABIDA), Mumbai	Shri K. L. Raphael Shri Kirti Gajjar (*Alternate*)
Godavari Biorefineries Ltd, Mumbai	Shri Shanul Laxmanrao Pagar Ms Wani A. J. (*Alternate*)
Gujarat Narmada Valley Fertilizers Company Limited, Gujrat	Dr M. J. Kapadia Shri P. R. Desai (*Alternate*)
Hindustan Organic Chemicals Ltd, (HOCL), Mumbai	Shri Deleep Kumar K. Shri V. Mohan (*Alternate*)
India Glycols Limited, Uttarakhand	Shri S. R. Soni Shri Alok Singhal (*Alternate*)
Laxmi Organic Indusrties, Mumbai	Shri J. P. Suryavanshi Dr Vijay S. Mishra (*Alternate*)
Ministry of Chemicals & Fertilizers, New Delhi	Shri O. P. Sharma Shri Varun Singh Poonia (*Alternate*)

Organization(s)	*Representative(s)*
National Test House, Ghaziabad	Shri Debashis Saha Dr Gopal Krishan (*Alternate*)
Reliance India Limited, Mumbai	Shri K. K. Sreeramachandran Shri Vasant Warke (*Alternate*)
BIS Director General, New Delhi	Shri N. K. Bansal, Scientist 'F' and Head (PCD), [Representing Director General (*Ex-Officio*)]

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Shri Chandrakesh Singh
Scientist 'D' (PCD), BIS

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